METHOD OF MAKING A THICK AND SMOOTH EMBOSSED TISSUE

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Cross-Reference to Related Application

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Field of the invention

The present invention relates to paper tissue products, such as, for example, facial tissue and disposable handkerchiefs. More particularly, the invention relates to converting steps for such tissue paper products, namely embossing and calendering.

Background of the invention

Paper webs or sheets, sometimes called tissue or paper tissue webs or sheets, and products made therefrom, such as paper handkerchiefs, sometimes also called facial tissues, find extensive use in modern society. Such items as facial and toilet tissues and kitchen towels are staple items of commerce, all of which are herein referred to as paper tissue products. It has long been recognized that important physical attributes of these products are their strength and thickness/caliper, their softness and smoothness, their absorbency, and their lint resistance. Research and development efforts have been directed to the improvement of each of these attributes without seriously affecting the others as well as to the improvement of two or three attributes simultaneously.

Softness and smoothness relate to the tactile sensation perceived by the consumer when holding a particular product, rubbing it across the skin, or crumpling it within the hands. The tactile sensation is a combination of several physical properties. The tactile sensation can be well captured by the objective parameter of the physiological surface smoothness (PSS) parameter as known e.g. from U.S. Patent No. 5,855,738. As important for the tactile sensation of consumers is the thickness/caliper of a tissue product.

Strength is the ability of the product to maintain physical integrity and to resist tearing, bursting, and shredding under use conditions.

Absorbency is the measure of the ability of a product to absorb quantities of liquid, particularly aqueous solutions or dispersions. Overall absorbency as perceived by the consumer is generally considered to be a combination of the total quantity of a liquid a given mass of paper tissue will absorb at saturation as well as the rate at which the mass absorbs the liquid.

Lint resistance is the ability of the fibrous product, and its constituent webs, to bind together under use conditions, including when wet. In other words, the higher the lint resistance is, the lower the propensity of the web to lint will be.

Relatively thick and yet soft disposable paper products, namely in the form of paper handkerchiefs and facial tissues, are known. For example, Tempo™, sold by The Procter & Gamble Company, is a four ply facial tissue paper product experienced as thick and soft and having a caliper of about 0.3 mm. A high caliper conveys the idea of high dry and wet strength to the consumer. A high wet strength, also referred to as wet burst strength, in particular prevents tearing or bursting which for a paper handkerchief in turn results in contamination of the user's hand with mucus or other bodily fluids.

In attempting to provide a very smooth surface it is common in the art to subject paper tissue to calendering. However, calendering generally means a trade-off of caliper and softness for smoothness (as discussed e.g. in U.S. Patent No. 5,855,738).

Through-air-dried tissue products generally exhibit relatively high wet burst strength and relatively high caliper versus conventional tissue. Though-air-drying facilities, however, are not generally available on conventional paper making machines and the provision of such equipment means a considerable financial investment. In a further aspect though-air-drying facilities have an increased energy consumption as compared to more conventional drying facilities. Therefore it is still of interest to provide superior paper qualities employing conventional paper machines.

Hence, there is a persisting challenge to provide a paper handkerchief satisfying or even excelling the standards of known products in meeting all relevant physical parameters without increasing the use of material or energy. Ideally, the same consumer benefit is provided using less cellulose raw material.

Accordingly, there remains a need for a tissue product, in particular a facial tissue, which:

- combines optimal strength (e.g. wet burst strength), absorbency and lint resistance.
- gives an improved tactile sensation of softness, smoothness and thickness.
- is cost effective to manufacture and can be manufactured on conventional paper machines as well as through-air-dried machines.

Summary of the Invention

The present invention relates to a paper tissue, and in particular to facial tissue, and disposable handkerchiefs. In particular, a method is described for making a tissue paper product from a tissue paper web, the method comprising the steps of:

- passing the tissue paper web through an embossing nip formed between a first and a second embossing roll, wherein at least one of the embossing rolls comprises at least 30 embossing elements per square centimeter; and
- passing the tissue paper web through a calendering nip formed between a first and a second calendering roll, wherein the first and the second calendering roll are in contact with the tissue paper web over a contact length measured parallel to the direction of the axis of the first calendering roll exert a pressure onto the paper web of at least about 50 N per centimeter of the contact length.

Further claimed are paper tissue products made in accordance with the above method.

Detailed Description of the Invention

Suitable papermaking steps

According to the present invention, a cellulosic fibrous structure is made using principles and machinery well-known in the art of paper-making. A suitable pulp furnish for the process of making the paper tissue substrate preferably contains papermaking fibers consisting essentially of cellulose fibers (commonly-known as wood pulp fibers) or cellulose-derived fibers (including, for example, rayon, viscose). Fibers derived from soft woods (gymnosperms or coniferous trees) and hard woods (angiosperms or deciduous trees) are contemplated for use in this invention. The particular species of tree from which the fibers are derived is immaterial. The wood pulp fibers can be produced from the native wood by any convenient pulping process. Chemical processes such as sulfite, sulphate (including the Kraft) and soda processes are suitable. Mechanical processes such as thermochemical (or Asplund) processes are also suitable. In addition, the various semi-chemical and chemi-mechanical processes can be used. Bleached as well as unbleached fibers are contemplated for use.

In addition to the above, other methods of making the tissue web may be employed and fibers and/or filaments made from polymers, for example hydroxyl polymers, may be used in the present invention. Nonlimiting examples of suitable hydroxyl polymers include polyvinyl

alcohol, starch, starch derivatives, chitosan, chitosan derivatives, cellulose derivatives, gums, arabinans, galactans and mixtures thereof.

The paper tissue according to the present invention may contain a wet strength chemical agent. Preferably up to about 3.0%, preferably at least about 0.5%, and more preferably at least about 0.8% by weight, on a dry fiber weight basis, of wet strength chemical agent, such as water-soluble permanent and temporary wet strength resin, are contained.

Wet strength resins useful herein can be of any type. For example, Westfelt described a number of such materials and discussed their chemistry in Cellulose Chemistry and Technology, Volume 13, at pages 813-825 (1979).

Usually, the wet strength resins are water-soluble, cationic materials. That is to say, the resins are water-soluble at the time they are added to the papermaking furnish. It is possible that subsequent events such as cross-linking will render the resins insoluble in water. Further some resins are soluble only under specific conditions, such as over a limited pH range. Wet strength resins are generally believed to undergo a cross-linking or other curing reactions after they have been deposited on, within, or among the papermaking fibers. Cross-linking or curing does not normally occur so long as substantial amounts of water are present.

Of particular utility are the various polyamide-epichlorohydrin resins. These materials are low molecular weight polymers provided with reactive functional groups such as amino, epoxy, and azetidinium groups. Descriptions of processes for making such materials, include U.S. Patent No. 3,700,623, issued to Keim on October 24, 1972, and U.S. Patent No. 3,772,076, issued to Keim on November 13, 1973.

Polyamide-epihydrochlorin resins sold under the trademarks Kymene 557H and Kymene LX by Hercules Inc. of Wilmington, Delaware, are particularly useful in this invention. These resins are generally described in the aforementioned patents to Keim.

Base-activated polyamide-epichlorohydrin resins useful in the present invention are sold under the Santo Res trademark, such as Santo Re 31, by Monsanto Company of St. Louis, Missouri. These types of materials are generally described in U.S. Patent No. 3,855,158 issued to Petrovich on December 17, 1974; U.S. Patent No. 3,899,388 issued to Petrovich on August 12, 1975; U.S. Patent No. 4,129,528 issued to Petrovich on December 12, 1978; U.S. Patent No. 4,147,586 issued to Petrovich on April 3, 1979; and U.S. Patent No. 4,222,921 issued to Van Eenam on September 16, 1980.

Other water-soluble cationic resins useful herein are the polyacrylamide resins such as those sold under the Parez trademark, such as Parez 631NC, by American Cyanamid Company of Sandford, Connecticut. These materials are generally described in U.S. Patent No. 3,556,932

issued to Coscia et al on January 19, 1971; and U.S. Patent No. 3,556,933 issued to Williams et al. on January 19, 1971.

Other types of water-soluble resins useful in the present invention include acrylic emulsions and anionic styrene-butadiene latexes. Numerous examples of these types of resins are provided in U.S. Patent No. 3,844,880. Meisel, Jr et al., issued October 29, 1974. Still other water-soluble cationic resins finding utility in this invention are the urea formaldehyde and melamine formaldehyde resins. These polyfunctional, reactive polymers have molecular weights on the order of a few thousand. The more common functional groups include nitrogen containing groups such as amino groups and methylol groups attached to the nitrogen. Polyethylenimine type resins also find utility in the present invention.

More complete descriptions of the aforementioned water-soluble resins, including their manufacture, can be found in TAPPI Monograph Series No. 29, "Wet Strength in paper and Paperboard, Technical Association of the Pulp and Paper Industry (New York; 1965).

Temporary wet strength agents, such as modified starch may also, optionally, be used as may combinations of permanent and temporary wet strength agents.

The present invention may contain dry strength chemical agents, preferably at levels up to about 3% by weight, more preferably at least about 0.1% by weight, on a dry fiber weight basis. A highly preferred dry strength chemical agent is carboxymethyl cellulose. Other suitable dry strength chemical agents include polyacrylamide (such as combinations of Cypro™ 514 and Accostrength™ 711 produced by American Cyanamid of Wayne, N.J.); starch (such as corn starch or potato starch); polyvinyl alcohol (such as Airvol™ 540 produced by Air Products Inc. of Allentown, PA); guar or locust bean gums; and polyacrylate latexes. Suitable starch materials may also include modified cationic starches such as those modified to have nitrogen containing groups such as amino groups and methylol groups attached to nitrogen, available from National Starch and Chemical Company (Bridgewater, NJ).

Chemical softening compositions, comprising chemical debonding agents are optional components of the present invention. U.S. Patent No. 3,821,068, issued June 28, 1974 teaches that chemical debonding agents can be used to reduce the stiffness, and thus enhance the softness, of a paper tissue web. U.S. Patent No. 3,554,862, issued on January 12, 1971 discloses suitable chemical debonding agents. These chemical debonding agents include quaternary ammonium salts.

Certain preferred chemical softening compositions comprise from about 0.01% to about 3.0% of a quaternary ammonium compound, preferably a biodegradable quaternary ammonium compound; and from about 0.01% to about 3.0% of a polyhydroxy compound; preferably

selected from the group consisting of glycerol, sorbitols, polyglycerols having an average molecular weight of from about 150 to about 800 and polyoxyethylene glycols and polyoxypropylene glycols having a weight average molecular weight from about 200 to 4000. Preferably the weight ratio of the quaternary ammonium compound to the polyhydroxy compound ranges from about 1.0:0.1 to 0.1:1.0. It has been discovered that the chemical softening composition is more effective when the polyhydroxy compound and the quaternary ammonium compound are first premixed together, preferably at a temperature of at least about 40°C, before being added to the papermaking furnish. Either additionally, or alternatively, chemical softening compositions may be applied to the substantially dry paper tissue web, for example by means of a printing process (N.B. all percentages herein are by weight of dry fibers, unless otherwise specified).

Examples of quaternary ammonium compounds suitable for use in the present invention include either unmodified, or mono- or di- ester variations of well-known dialkyldimethylammonium salts and alkyltrimethyl ammonium salts. Examples include the di-ester variations of di(hydrogenated tallow)dimethyl ammonium methylsulphate and di-ester variations of di(hydrogenated tallow)dimethyl ammonium chloride. Without wishing to be bound by theory, it is believed that the ester moity(ies) lends biodegradability to these compounds. Commercially available materials are available from Witco Chemical Company Inc. of Dublin, Ohio, under the tradename "Rewoquat V3512". Details of analytical and testing procedures are given in WO95/11343, published on 27th April, 1995.

Examples of polyhydroxy compounds useful in the present invention include polyoxyethylene glycols having a weight average molecular weight of from about 200 to about 600, especially preferred is "PEG-400".

While the addition of particular chemical agents listed above as preferred agents, can have very beneficial effects on the paper products obtained, namely their softness, paper tissue webs useful for the present invention may be made by any common method well-known to the person skilled in the art.

Such papermaking processes may comprise the dewatering of suitable pulp using, for example, one or more papermakers felts and/or belts. Conventional paper making processes can be used. Any process referred to herein as conventional is a paper-making process which does not comprise a step of through-air-drying. Alternatively, papermaking processes comprising a through-air-drying step can be utilized.

<u>Stretch embossing step</u>

The present invention is specifically concerned with steps known in the art as converting steps. One important converting step to be carried out in accordance with the present invention is an embossing step in which a very fine pattern is embossed using a low pressure.

Embossing of a paper tissue web is generally achieved by passing the web through the nip formed between two embossing rolls, at least one embossing roll comprising embossing elements. An embossing roll typically comprises a curved, but otherwise flat surface. Embossing elements are protrusions raising above this surface and having a certain height as measured in a direction perpendicular to the axis of the embossing roll from the curved flat roll surface to the utmost point of the protrusion. Embossing elements have a certain width, to be measured in the plane of the essentially flat roll surface. The term width as used herein refers to the diameter of a round embossing element measured the plane specified above (i.e. at the bottom of the embossing element) or to the largest width measured in said plane, when the embossing element is not round.

According to the present invention the embossing elements can have any shape, such as pyramidal or half spherical, and the cross section of the embossing elements can be circular, oval or square. The embossing elements may form a continuous pattern, but preferably are distinct form each other. The embossing elements may be disposed over at least one embossing roll in a very fine pattern, comprising at least about 30 embossing elements, preferably at least about 50, more preferably at least about 60, yet more preferably at least about 70, most preferably at least about 80 embossing elements per square centimeter surface area of the embossing roll.

According to the present invention the embossing elements are not high, preferably they have a height of less than about 1 mm, more preferably less than about 0.8 mm, yet more preferably less than about 0.6 mm, yet even more preferably less than about 0.5 mm or less than about 0.4 mm, and most preferably less than about 0.3 mm.

Preferably the stretch embossing provides a ratio of embossed areas to unembossed areas from about 5% - 95%, more preferably about 20% to about 80% and most preferably about 40% to about 60%, i.e. for the most preferred case about 40% to about 60% of the total surface area of the tissue paper web are embossed.

Any known type of embossing roll and mode of operation of such roll is within the scope of the present invention. In one preferred embodiment of the present invention two hard metal (e.g. steel), embossing rolls are used, wherein a first roll comprises protruding embossing elements, referred to as the male roll, and a second roll comprises matching recesses, referred to a the female roll. The recesses may be mirror images of the protruding embossing elements or

may be adapted to be slightly smaller than exact mirror images, e.g. due to a slight difference in size or shape (e.g. slope) of those recesses in the female roll.

In another embossing step according to the present invention a first embossing roll comprises a web contacting surface provided from a hard material comprising protruding embossing elements and a second roll comprises a web contacting surface comprising a softer material, e.g. rubber, preferably a material of Shore A hardness 40-70, in which recesses are formed upon sufficiently close contact with the protruding embossing elements. Providing an embossing nip from a hard roll in combination with a rubber roll has numerous advantages, such as cheaper and easier production and operation, since the adjustment of the rolls in much less critical than for a male and a female hard roll. Surprisingly, it has been found that the method claimed herein also provides excellent results when a hard rubber roll combination is used.

The size of the nip formed between the two embossing rolls is to be adapted depending e.g. on the tissue paper web to be processed and depending on the embossing pattern used. Also depending on those considerations no pressure or some pressure may be applied to urge the first embossing roll and the second embossing roll together.

When two hard rolls are employed in the process, a male and a female role, the rolls should be operated so as to leave a space corresponding to about 60% to 140%, preferably about 80% to about 120% of the caliper of the unembossed tissue paper between the protruding embossing elements of the male role and the bottom of the recesses of the female role.

When a hard roll is used in combination with a rubber roll, the rolls should be pressed against each other with a pressure of about 10 N/square centimeter to about 1000 N/square centimeter, preferably about 20 N/square centimeter to about 200 N/square centimeter and most preferably about 50 N/square centimeter to about 100 N/square centimeter.

Known modes of operation are suitable for the present invention. In certain embodiments, the embossing rolls are not heated and run at the same speed, but in alternative modes of operation at least one roll may be heated and the rolls may run at unequal speed.

The above described embossing with a fine pattern, in one important aspect serves to increase the caliper, or in other words the bulk of the paper tissue web. Therefore, in one embodiment of the present invention, a single web or a single ply of paper tissue is passed through the embossing nip. In alternative modes of operation a multitude of plies of paper may be passed through the nib at the same time. The method may also employ a separate and distinct joining step as to provide a multiply tissue paper product, the joining step preferably comprising an embossing step, such as "attachment embossing" described hereinafter.

Calendering step

Any known method of calendering can be employed in the converting process, however, in accordance with the present invention unusually high calendering pressures are used.

A calendering step in accordance with the present invention may comprise passing one or several tissue paper webs through a calendering nip formed between a first and a second calendering roll. Typically both calendering rolls contact the web over a certain length, herein referred to a contact length, measured parallel to the direction of the axis of said first calendering roll. The calendering rolls exert a pressure onto the web of at least about 30 N per centimeter of said contact length and in order to do so will be pressed against each other with such a pressure. More preferably the pressure per centimeter of said contact length is from about 50 N to about 300 N, more preferably about 60 N to about 250 N, yet more preferably about 70 N to about 200 N and most preferably about 120 N to about 150 N. According to the present invention preferably as many paper tissue webs are calendered as the paper tissue product will comprise plies, for example two, three or four webs can be juxtaposed and calendered in one step.

Known equipment and known modes of operation are suitable for the present invention. In certain embodiments, the calendaring rolls are not heated and run at the same speed, but in alternative modes of operation at least one roll may be heated and the rolls may run at unequal speed.

Calendering is well known in the art to reduce the caliper of a tissue paper web, and typically employed to ensure the caliper of the paper tissue product meets the required specifications. However, due to the pressure employed, leading to a densification of the paper web, calendering is typically known to reduce the perceived softness of a paper tissue product. Calendering is therefore, at least in the area of hygiene papers, such a paper handkerchiefs, carried out at not too high pressures, typically for an embossed paper web about 10 N/cm to about 20 N/cm are selected.

It has been surprisingly found that the embossing step in combination with the calendering step leads to a rather thick and bulky and yet still very soft paper product. More particularly, it has been found that the paper tissue web after undergoing a stretch embossing step and a calendering step is of increased caliper as compared to the untreated web. (When e.g. three webs are calendered in one step the comparison is to be made between three layers of untreated web versus three layers of embossed and calendered web.) This effect is particularly surprising, a calendering with a high pressure is known to reduce the caliper of a paper web considerably.

The method claimed in the present invention has been found to increase the caliper of a paper tissue web by about 10%, sometimes even about 30% and even up to about 40%, about 60%, about 80% or about 100% when comparing the caliper of the untreated web with the caliper of the treated web. The stretch embossing step alone achieves caliper increases of typically about 50% to about 200%.

A paper tissue according to the present invention has a first and a second surface, the surfaces being mutually opposed to each other, and a thickness orthogonal to the first and second surface. The thickness is also referred to a caliper of the tissue. The caliper of a 3-ply paper tissue product according to the present invention is preferably from about 0.1 mm to about 1 mm, more preferably from about 0.2 mm to about 0.5 mm.

Moreover, a paper tissue according to the present invention has preferably a wet burst strength greater than about 50g, more preferably greater than about 100 g, preferably from about 150 g to about 500 g, more preferably from about 250 g to about 400 g.

It has been found that the method claimed herein leads to a considerable reduction of the dry tensile strength of the paper tissue without seriously affecting the wet tensile strength of the paper tissue. Paper tissues treated with the claimed method typically achieve a dry tensile strength from about 1000g to about 2500g and a wet burst strength of about 100 g to about 300 g and preferably achieve a dry tensile strength to wet burst strength ratio of about 0.1 to about 0.3, preferably about 0.125 to about 0.25 and most preferably about 0.15 to about 0.2.

In a further aspect, a paper tissue product according to the present invention preferably has a physiological surface smoothness parameter of less than about 1000 microns, preferably from about 650 microns to about 50 microns, more preferably from about 650 microns to about 300 microns.

In one preferred embodiment of the present invention a paper tissue product is provided from two plies to four plies, three plies being most preferred. Preferably all plies comprise a stretch embossing pattern extending over at least about 50%, but preferably about 80% of the whole surface area of the paper tissue product and most preferably the whole surface area of the paper tissue product.

<u>Other process steps</u>

The method for making a tissue paper product according to the present invention may comprise a number of further optional steps. For example, a lotion may be applied by any suitable means, such as printing or spraying. The lotion can either be applied to the paper web or a paper tissue product, either to the whole surface of the web or product or only to a portion

thereof. For a multiple ply paper tissue product the lotion may be applied to all plies or only selected plies and to only one or to both surfaces of the plies. In one preferred embodiment lotion is applied to both outer surfaces of the paper tissue product.

A lotion has been found to contribute to the smoothness of the paper tissue, and hence decrease its PSS parameter. Moreover, the lotion may have skin care benefits.

The lotion may comprise softening/debonding agents, emollients, immobilizing agents and mixtures thereof. Suitable softening/debonding agents include quaternary ammonium compounds, polysiloxanes, and mixtures thereof. Suitable emollients include propylene glycol, glycerine, triethylene glycol, spermaceti or other waxes, petrolatum, fatty acids, fatty alcohols and fatty alcohol ethers having from 12 to 28 carbon atoms in their fatty acid chain, mineral oil, namely silicone oil e.g. dimethicone and isopropyl palmitate, and mixtures thereof. Suitable immobilizing agents include ceresin, stearyl alcohol and paraffins, polyhydroxy fatty acid esters, polyhydroxy fatty acid amides, and mixtures thereof.

Other optional components include perfumes, antibacterial actives, antiviral actives, disinfectants, pharmaceutical actives, film formers, deodorants, opacifiers, astringents, solvents and the like. Particular examples of lotion components include camphor, thymol, menthol, camomile extract, aloe vera, calendula officinalis.

Certain lotions according to the present invention are highly transferable lotions comprising at least some of the components listed above, as transferability helps to ensure superior skin care and pharmaceutical benefits.

Juxtaposed plies of the paper tissue web may be joined as to provide a multi ply paper tissue product, preferably by attachment embossing. "Attachment embossing", as used herein, refers to an embossing by which all plies of a multi-ply tissue according to the present invention are embossed in one process step. Preferably the attachment embossing does not or at least not to a large extent affect the smoothness of any calendered ply. Therefore, preferably the tissue has an unembossed surface over a major part of the surface area of the tissue, preferably on the first and the second surface. As used herein, this means that the tissue has one or more regions not comprising an attachment embossing and, optionally, one or more regions comprising an attachment embossing, and that the region not comprising an attachment embossing is at least about 50%, preferably at least about 80% and in some preferred embodiments as much as about 99%, of the surface area of the tissue. Most commonly the regions comprising an attachment embossing lie close to the edge of the tissue (for example along two or four edges); and a regions comprising an attachment embossing may also be used for decorative purposes (for example to create a pattern or to spell out a logo or brand name). The region not comprising an attachment

embossing is the continuous region between and/or around the region comprising an attachment embossing.

Attachment embossing is preferably done by pin-to-pin embossing and with about 10 to about 40 embossing elements per square centimeter having a height from about 0.01 mm to about 1 mm, preferably about 0.05 mm to about 0.2 mm. The percentage of attachment embossed areas to unembossed or fine embossed areas of the total surface area of a paper tissue product is preferably about 0.01% to about 5%. Attachment embossing involves as substantive densification of the paper tissue products as to achieve the attachment. Therefore the space between and embossing element and its counterpart, e.g. two pins where pin-to-pin embossing is employed, is less that the caliper of the paper tissue to be embossed, typically about 5% to about 50%, preferably about 10% to about 20% of the caliper of the paper tissue to be embossed, which leads to embossing pressures of about 10 000 to about 50 000 N/square centimeter.

The method of the present invention may further comprise a step of providing sheets suitable for paper tissue products, such as paper handkerchiefs. Such step typically comprises cutting of portions of the paper tissue web.

If desired, the paper tissue products according to the present invention may be provided with functional or aesthetic indicia. The indicia may be applied to either or both of the surfaces of the paper tissue products. The indicia may cover all or part of the paper tissue products and be applied in a continuous or discontinuous pattern.

The indicia may be applied to the paper tissue products by any means well known in the art, such as spraying, extruding, and preferably printing. Examples of printing methods include gravure or flexographic printing. If printing is selected as the means for applying the indicia, the printing apparatus may be constructed according to the teachings of commonly assigned U.S. Patent No. 5,213,037 issued May 25, 1993 to Leopardi, II. If desired, the apparatus may have reservoir baffles, as disclosed in commonly assigned U.S. Patent No. 5,255,603 issued October 26, 1993 to Sonneville et al. If desired, the indicia may be requested with perforations or drop off cuts as disclosed in commonly assigned U.S. Patent No. 5,802,974 issued Sept. 8, 1998 to McNeil. The disclosures of the aforementioned patents are incorporated herein by reference.

Test Methods

Caliper is measured according to the following procedure: The tissue paper is preconditioned at 21° to 24°C and 48 to 52 percent relative humidity for two hours prior to the caliper measurement. If the caliper of toilet tissue is being measured, 15 to 20 sheets are first removed and discarded. If the caliper of facial tissue is being measured, the sample is taken from

near the center of the package. The sample is selected and then conditioned for an additional 15 minutes.

Caliper of the multi-ply paper tissue, as used herein, is the thickness of the paper when subjected to a compressive load of 14.7 g/cm^2. Preferably, caliper is measured using a low load Thwing-Albert micrometer, Model 89-11, available from the Thwing-Albert Instrument Company of Philadelphia, Pa. The caliper per ply is the total caliper of the multi-ply paper tissue divided by the number of plies comprised. For a single ply tissue caliper per ply and caliper are identical. Decorated regions, perforations, edge effects, etc., of the tissue should be avoided if possible.

The <u>wet burst strength</u> is measured using an electronic burst tester and the following test conditions. The burst tester is a Thwing-Albert Burst Tester Cat. No. 177 equipped with a 2000 g load cell. The burst tester is supplied by Thwing-Albert Instrument Company, Philadelphia, PA 19154, USA.

Take eight paper tissues and stack them in pairs of two. Using scissors, cut the samples so that they are approximately 228 mm in the machine direction and approximately 114 mm in the cross-machine direction, each two finished product units thick.

First age the samples for one to two hours by attaching the sample stack together with a small paper clip and "fan" the other end of the sample stack to separate the sheets, this allows circulation of air between them. Suspend each sample stack by a clamp in a 107°C (± 3°C) forced draft oven for 5 minutes (± 10 seconds). After the heating period, remove the sample stack from the oven and cool for a minimum of three minutes before testing.

Take one sample strip, holding the sample by the narrow cross direction edges, dipping the center of the sample into a pan filled with about 25mm of distilled water. Leave the sample in the water four (4.0 ± 0.5) seconds. Remove and drain for three (3.0 ± 0.5) seconds holding the sample so the water runs off in the cross direction. Proceed with the test immediately after the drain step. Place the wet sample on the lower ring of the sample holding device with the outer surface of the product facing up, so that the wet part of the sample completely covers the open surface of the sample holding ring. If wrinkles are present, discard the sample and repeat with a new sample. After the sample is properly in place on the lower ring, turn the switch that lowers the upper ring. The sample to be tested is now securely gripped in the sample holding unit. Start the burst test immediately at this point by pressing the start button. The plunger will begin to rise. At the point when the sample tears or ruptures, report the maximum reading. The plunger will automatically reverse and return to its original starting position. Repeat this procedure on

three more samples for a total of four tests, i.e., 4 replicates. Report the results, as an average of the four replicates, to the nearest gram.

The dry tensile strength is measured according to the following procedure: The test is performed on one inch by five inch (about 2.5 cm X 12.7 cm) strips of paper (including handsheets as described below, as well as other paper sheets) in a conditioned room where the temperature is 28°C + 2.2°C and the relative humidity is 50% + 10%. An electronic tensile tester (Model 1122, Instron Corp., Canton, Mass.) is used and operated at a crosshead speed of 2.0 inches per minute (about 5.1 cm per min.) and a gauge length of 4.0 inches (about 10.2 cm). Reference to a machine direction means that the sample being tested is prepared such that the 5" dimension corresponds to that direction. Thus, for a machine direction (MD) dry tensile strength, the strips are cut such that the 5" dimension is parallel to the machine direction of manufacture of the paper product. For a cross machine direction (CD) dry tensile strength, the strips are cut such that the 5" dimension is parallel to the cross-machine direction of manufacture of the paper product. Machine-direction and cross-machine directions of manufacture are well known terms in the art of paper-making. The MD and CD tensile strengths are determined using the above equipment and calculations in the conventional manner taking the arithmetic average of at least six strips tested for each directional strength. The dry tensile strength, as used herein, is the arithmetic average of the average MD and the average CD tensile strengths.

For the physiological surface smoothness measurement, which reports the PSS parameter, a sample of the paper tissue is selected which avoids wrinkles, tears, perforations, or gross deviations from macroscopic monoplanarity. The sample is conditioned at 22 to 24°C and 48 to 52% relative humidity for at least two hours prior to testing. The sample is placed on a motorized table and magnetically secured in place. Either face of the sample may be selected for the measurement, provided all traces are taken from the same face.

Physiological surface smoothness is obtained by scanning the paper tissue sample in any direction with a profilometer to obtain the Z-direction displacement as a function of distance. The Z-direction displacement is converted to an amplitude versus frequency spectrum by a Fourier Transform. The spectrum is then adjusted for human tactile response using a series of filters. The peak heights of the filtered amplitude frequency curve are summed from 0 to 10 cycles per millimeter to give the result.

The paper tissue sample is approximately 100 millimeters x 100 millimeters in size and mounted on a motorized table. While any suitable table will suffice, a table with surface tester model KES-FB-4NKES-SE, available from Kato Tech Company Limited of Koyota, Japan, or a CP3-22-01 DCI Mini Precision table using a NuStep 2C NuLogic Two Axis Stepper Motor

Controller in the closed loop control mode have been found suitable. The table has a constant drive motor which travels at the rate of 1 millimeter per second. The sample is scanned 30 millimeters in the forward direction transversely indexed one millimeter, then reversed. Data are collected from the center 26 millimeters of the scan in both the forward and reverse directions. The first and last 2 millimeters of each scan are ignored and not used in the calculations.

The profilometer has a probe with a tip radius of 2.54 microns and an applied force of 0.20 grams. The gauge range is calibrated for a total Z-direction displacement of 3.5 millimeters. Over the scan distance of the sample, the profilometer senses the Z-direction displacement of the stylus in millimeters. The output voltage from the gauge controller is digitized at a rate of at least 20 points per second. Over the entire 26 millimeter scan range, 512 pairs of time surface height data points are obtained for both the forward and reverse directions of a scan. The profilometer is mounted above the sample table such that the surface topography can be measured. A suitable profilometer is a EMD 4320 WI Vertical Displacement Transducer, having an EPT 010409 stylus tip, and an EAS 2351 Analog Amplifier. This equipment is obtainable from Federal Products of Providence, Rhode Island.

The digitized data pairs are imported into a standard statistical analysis package for further analysis. Suitable software analysis packages included SAS of Cary, North Carolina, and preferably LabVIEW Instrument Control Software 3.1 available from National Instruments of Austin, Texas. When using the LabVIEW software, raw data pairs linking surface height and time from the individual scans are centered about the mean using the Mean.vi analysis tool in the LabVIEW software. The 512 data points from each of the 16 traces are converted to 16 amplitude spectra using the Amplitude and Phase Spectrum.vi tool. Each spectrum is then smoothed using the method described by the PROC Spectra Method of the SAS software. LabVIEW smoothing filter values of 0.000246, 0.000485, 0.00756, 0.062997, 0.00756, 0.000485, 0.000246 are utilized. The output from this tool is taken as the Amp Spectrum Mag (vrms).

The amplitude data are then adjusted for human tactile response using a series of frequency filters designed from Verrillo's data on vibrotactile thresholds as a function of vibration frequency as set forth in the Journal of Acoustical Society of America, in the article entitled "Effect Of Contactor Area On The Vibrotactile Threshold", Vol. 35, 1962 (1963). The aforementioned data are reported in a time domain as cycles per second and converted to the spatial domain in cycles per millimeter. The conversion factor and filter values are found in the procedure set forth in the 1991 International Paper Physics Conference, TAPPI Book 1, more particularly the article entitled "Methods For The Measurement Of The Mechanical Properties

Of Paper tissue" by Ampulski, et al., and found at page 19, utilizing the specific procedure set forth at page 22 entitled "Physiological Surface Smoothness". The response from the filters are set at 0 below the minimum threshold and above the maximum response frequencies and varies from 0 to 1 therebetween as described by the aforementioned Ampulski et al. article.

The physiologically adjusted frequency amplitude data are obtained by multiplying the amplitude spectra described above by the appropriate filter value at each frequency. A typical amplitude spectrum and filtered amplitude spectrum are illustrated in Fig. 5 of the aforementioned Ampulski et al. article. The Verrillo-adjusted frequency amplitude curve is summed point by point between 0 and 10 cycles per millimeter. This summation is considered to be the physiological surface smoothness. The eight forward and eight reverse physiological surface smoothness values thus obtained are then averaged and reported in microns.

Physiological surface smoothness measurements using the SAS software is described in commonly assigned U.S. Patent Nos. 4,959,125, issued Sept. 25, 1990 to Spendel; 5,059,282, issued Oct. 22, 1991 to Ampulski et al.; 5,855,738, issued Jan. 5, 1999 to Weisman et al., and 5,980,691, issued Nov. 9, 1999 to Weisman et al.

Either face of the tissue may be selected for the smoothness measurement, provided all traces are taken from the same face. If either face of the tissue meets any of the smoothness criteria set forth herein, the entire sample of the tissue is deemed to fall within that criterion. Preferably both faces of the tissue meet the above criteria.

<u>Example</u>

An aqueous slurry comprising 3% by weight of Nothern Softwood Kraft (NSK) fibers is prepared in a conventional re-pulper. The NSK slurry is refined gently and a 2% solution of the permanent wet strength resin (Kymene™ 617) is added to the NSK stock pipe at a rate of 0.9% by weight of the total dry fibers. The absorption of the permanent wet strength resin onto the NSK fibers is enhanced by an in-line mixer. A 1% solution of the dry strength resin (carboxymethyl cellulose) is added to the NSK stock before the fan pump at a rate of 0.14% by weight of the total dry fibers. The NSK slurry is diluted to about 0.2% consistency at the fan pump.

A chemical softening composition is prepared comprising di-hard tallow diethyl ester dimethyl quaternary ammonium chloride and polyoxyethylene glycol, having an average molecular weight of 400 (PEG-400). The PEG-400 was heated to about 66°C, and the quat is dissolved into the molten PEG-400 so that a homogeneous mixture is formed.

An aqueous slurry comprising 3% by weight of eucalyptus fibers is prepared in a conventional re-pulper. A 1% solution of the chemical softening composition is added to the Eucalyptus stock pipe at a rate of 0.09% by weight of the total dry fibers. The Eucalyptus slurry is diluted to about 0.2% consistency at the fan pump. The 1% solution of the chemical softening composition is also added to the NSK slurry after post CMC addition and prior to dilution of the slurry to about 0.2% at the stock pump.

The two slurries were combined so that the ratio of NSK to eucalyptus fibers is 40:60 and the resulting slurry is deposited, by means of a single layer headbox onto a Fourdrinier wire to form an embryonic web. Dewatering occurs through the Fourdrinier wire and is assisted by a deflector and vacuum boxes.

The embryonic web is transferred from the Fourdrinier wire, at a fiber consistency of about 20% at the point of transfer, to a conventional drying felt. The web is then transferred to the surface of a Yankee dryer with a sprayed creping adhesive comprising 0.25% aqueous solution of Polyvinyl Alcohol (PVA). The fiber consistency is increased to an estimated 96% before dry creping the web with a doctor blade. The doctor blade has a bevel angle of about 25° and is positioned with respect to the Yankee dryer to provide an impact angle of about 81°. The Yankee dryer is operated at about 4 m/s and the dried, uncalendared paper is formed into 1ply rolls at a reel.

Three of these 1-ply rolls are taken to an off-line rewinding operation to form 3-ply rolls that are subsequently converted into a 3-ply tissue paper product, having overall dimension of about 210 mm square.

The 3-ply rolls are produced by simultaneously unwinding 3 of the 1-ply rolls. While unwinding the paper tissue webs of the 1-ply rolls are each passed through an embossing nip formed between a hard rubber (Shore A hardness 60) and a steel roll, the steel roll comprising 80 oval embossing elements, 0.26 mm high. Subsequently the paper tissue webs are juxtaposed by a set of rolls, so that three juxtaposed webs are passed through a calendering nip formed between two steel calendering rolls, which are pressed against each other with a pressure of 160 N per cm contact length corresponding to a total pressure of 13440 N.

The 3-ply roll is subsequently converted into a 3-ply tissue product. The three ply web is unwound and subjected to an embossing step before folding. The margin of the tissue paper product, extending about 15mm in from the edge is embossed following the process described in WO95/27429, published on 19 October 1995. The major part of the surface area of the tissue paper product (i.e. all of the surface area within the 15mm margin) is unembossed. The tissue is

further decorated by embossing the brand name over a small area of the previously unembossed area and four decorative leaf patterns are embossed in the previously unembossed area.

Lotion is printed on each of the outer surfaces of the 3-ply web via a two step application process before folding. The lotion is an aqueous solution of di-hard tallow diethyl ester dimethyl quaternary ammonium chloride. The printing is accomplished by running the 3-ply web through two consecutive printing stations each consisting of an engraved anilox roll and a rubber backing roll pair.

The anilox roll is engraved to a cell volume of about 3 ml per square meter, and with supplied with lotion from a closed supply chamber designed to fill the engraved volume with lotion. A gap of 0.35mm is established between the anilox roll and backing roll, and the 3-ply web is run through this gap, transferring lotion to the surface touching the anilox roll. The web is then run through the second printing station with an identical anilox/rubber roll pair at a 0.35mm gap. The pairs are arranged such that the second anilox roll contacted the as yet unlotioned surface, transferring lotion to it. This arrangement transfers about 0.45% active quat per dry weight of the finished 3- ply tissue.

The paper tissue obtained by the above described process has a basis weight of 54 g/m^2, a total caliper of 0.35 mm, a caliper per ply of 0.12 mm, a wet burst strength of 250 g and a PSS parameter of 620 micron.

All documents cited are, in relevant part, incorporated herein by reference; the citation of any document is not to be construed as an admission that it is prior art with respect to the present invention.

While particular embodiments and/or individual features of the present invention have been illustrated and described, it would be obvious to those skilled in the art that various other changes and modifications can be made without departing from the spirit and scope of the invention. Further, it should be apparent that all combinations of such embodiments and features are possible and can result in preferred executions of the invention. Therefore, the appended claims are intended to cover all such changes and modifications that are within the scope of this invention.